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(54) Title: TYRE PUNCTURE SEALANT COMPOSITIONS

(57) Abstract

A liquid composition is provided which is used for "self-sealing" of punctures in tubed or tubeless vehicle tyres after a tyre is inadvertently punctured. The liquid composition contains 0.7 to 5 % fibre or particle filler, 0.5 to 15 % colloidal silica, 4 to 30 % glycol, from 1 to 8 % tackifier and 0.3 to 5 % surfactant, with the balance being water, providing the composition has a working viscosity (a viscosity as found in an operating vehicle tyre) of less than 20 cps. All percentages are by weight. The method of preventing inadvertent loss of air from pneumatic tyres by incorporating such a composition within the tyre is also claimed.

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TYRE PUNCTURE SEALANT COMPOSITIONS

This invention relates to a tyre puncture sealant composition, useful for "self-sealing" of punctures in tubed or tubeless vehicle tyres after a tyre is inadvertently punctured.

As is well known by automotive engineers, a tyre blow-out caused by puncturing of a tyre when a vehicle is in motion can be extremely dangerous and in some instances can result in loss of control of the vehicle with injury or loss of life resulting to the occupants of the vehicle.

Also, tyres which slowly go flat as a result of an inadvertent puncture in the tyre, can also cause substantial inconvenience due to the unreliability in vehicle operation, and the potential immobility of the vehicle for an indefinite period which results.

Several compositions are currently available which attempt to tackle the problem of inadvertent tyre puncture, by including a flowable composition within the vehicle tyre which moves within the tyre as it rotates and acts to reduce the size of any puncture in the tyre, whereby to prevent sudden loss of air therefrom.

However, numerous problems and deficiencies exist with such known compositions at this time. These problems include an un-balancing of the wheel to which the composition is added, so that upon rotation of the wheel during vehicle movement, unwanted vibrations are transmitted to the steering and/or suspension of the vehicle, with deleterious effects. This appears to be due to the large volume of composition which must be employed, to produce an acceptable sealing or restriction of the puncture in the tyre and the inability of the compositions to achieve a low viscosity.

Also, sealing of a puncture with these known compositions is often not achieved and air continues to escape through the puncture, so that while a complete "blowout" may be avoided, the tyre will go flat thereafter.

This latter consideration is more relevant for slow moving vehicles such as earth moving equipment and other large tyre vehicles.

It is an object of the present invention to provide a liquid composition to be placed within a tubed or tubeless tyre, which does not impart any significant un-balancing problems to an already balanced rim and tyre, upon movement of the vehicle.

It is also an object of the invention to provide a composition which more successfully seals an inadvertent puncture against loss of air from within the tyre, than previously known compositions.

In the broadest aspect of the invention there is provided a composition which contains from 0.7% to 5% filler; 0.5 to 15% silica in a colloidal form; from 4 to 30% of a glycol; from 1% to 8% tackifier which may also exhibit surfactant properties but where it does not act as a surfactant, from 0.3% to 5% surfactant; with the balance being water; said composition having a working viscosity (as hereinafter defined) of less than 20 cps.

All percentages referred to throughout the specification are percentages by weight.

Reference throughout the specification to a "working viscosity" means the viscosity of the composition when in a moving vehicle tyre under normal operating conditions.

All viscosities referred to in the specification are those as measured on a Brookfield viscometer, model DV-II, spindle 1, 12 rpm at 20°C. As will be appreciated by those skilled in the art the working viscosity in accordance with the invention can be closely approximated by vigorously shaking a test sample of the composition for 30 seconds or more and immediately testing the viscosity of the composition thereafter.

Accordingly, in a first preferred embodiment of the invention compositions of the invention may also include any one or more of the following additional components; up to 3% of a stabilized latex

solution; up to 20% of a water misable, polar alcohol; up to 5% of a mixture of two or more polyuronites either with or without impurity amounts of calcium oxalate; up to 1.5% PVA polymer; and up to 3% lecithin or linseed oil.

The filler used in this invention may be one or more of synthetic fibers, vegetable fibers and mineral or comminuted or finely divided rubber particles.

The synthetic or vegetable fibers are preferably between 0.5 and 6mm in length and between 12 and 32 microns in diameter. The only requirement for the fiber in addition to the physical parameters is that it is of a material compatible with the remainder of the solution which makes up the composition of the invention.

The mineral particles used for the invention are preferable diatomaceous earth particles and when present a suspension agent for the diatomaceous earth may be present. The suspension agent includes a polysaccaride gum such as xantham gum or guar gum and a metal aluminosilicate.

The comminuted or finely divided rubber particles are finally ground and are preferable of the order of 400 microns in diameter.

The total filler content in the composition of the invention may be made up from one or more of these four components.

The colloidal silica component of the invention is preferably made by combustion of silica tetrachloride in hydrogen-oxygen furnaces. The silica product is available in various hydrophilic and hydrophobic grades and although a single grade of one type of product may be employed in the invention, it has been found that a mixture of hydrophilic to hydrophobic in the ratios between 1:30 to 20:1 provides preferable results.

The glycol of the composition is preferably an alkyl or alkylene glycol inert to rubber or rubber containing compounds, and more particularly is inert to the rubber compounds from which

vehicle tyres are made. The alkyl or alkylene component of the glycol is preferable one having 5 to 25 carbon atoms therein.

The tackifier of the invention is required to provide some "tack" characteristic to the compositions of the invention. While a large number of known tackifiers may be employed, preferred tackifiers include acrylic polymers or acrylamide/acrylate copolymers, stabilised latex solutions and, PVA polymer solutions. Some tackifiers also exhibit surfactant characteristics to a sufficient degree to provide adequate wetting properties to the composition. Where this is not the case then the compositions of the invention require a surfactant to be added.

While many surfactants are commercially available which all have the necessary surfactant properties, preferred surfactants are of the non-ionic variety and may be one or more of the surfactants sold by ICI Chemicals Limited under the trade mark Teric.

The most particularly preferred range of viscosity compositions of the invention is from 2 cps to 10 cps.

As indicated previously this invention provides puncture sealing compositions which are effective over a wide range of types of vehicle tyres. Different types of tyres, may require different formulation techniques for the compositions and also the addition of one or more of the above referenced optional components to provide an enhanced performance under the particular conditions of use of the tyre.

As will be appreciated by those skilled in the art all compositions of the invention may include rust inhibitors, oxygen scavengers, and/or anti-fungal and anti-bacterial preservatives.

In a further preferred embodiment in the invention the pH of compositions included in the invention is adjusted to fall in the range of 8.0 to 8.5 inclusive.

Preferably the amount of PVA present is in the range of 0.1 to 1.5%.

The colloidal silica is preferably present in the amount of .5 to 10% by weight.

Lecithin, mentioned previously, also acts as a stabiliser for the latex solution when that is present. Other stabilisers may include one or more pH adjusting compounds such as ammonia and may also include silicone emulsions.

The volume of the compositions of the invention required for each tyre will depend on a number of factors, as will be appreciated by those skilled in the art. These factors include the diameter of the tyre and the width of the tyre.

Formulation of the composition may be achieved by admixture of the components, or by a pre-mixture of some components and a blending of the pre-mixtures to form the final composition. As will be appreciated by the person skilled in the Art, the various characteristics of the chosen components may indicate one method of formulation as opposed to another.

The following examples illustrate particularly preferred embodiments of aspects of the invention.

EXAMPLE I

A composition was formulated by mixing together the following compounds:

	Percentage by weight
Filler (synthetic viscose fibre)	1.0
Hexylene glycol	4.0
Colloidal silica	1.0
Tackifier (Acrylic latex available from RLA Polymers, Victoria, Australia under the product ref RL3034)	2.0
Surfactant (Teric LA8 available from ICI Chemicals)	0.5

Guar gum	0.1
Water	to 100%

To test the puncture sealing effectiveness of this composition 120 ml was inserted into a deflated tyre (Pirelli P205/65/R15 tubeless) through the valve inlet with the valve removed. The tyre was then inflated to 2.1 Kpa (32 psi).

Four steel nails 30mm X 2mm diameter were driven through the tread of the tyre and the tyre spun. The tyre immediately sealed around each nail and a check of the air pressure in the tyre showed no appreciable loss of air.

A test procedure was commenced using a standard builder's hammer. The puncture was struck with firm blows from the hammer until ten blows had occurred. If the seal around the puncture does not break the test is considered passed.

In this case, the seal on the punctures did not break after the 10 blows.

The tyre was then placed on a free spin axle and a rotating drum was placed against the tread. Pressure is applied to the drum while it is rotated to simulate normal road conditions.

The tyre was tested for 48 hours at the equivalent of 100 kph. At the end of this time the test was concluded and a check of the presence in the tyre showed that no appreciable air loss had occurred.

EXAMPLE II

A composition was formulated by mixing together the following compounds.

	Percentage by weight
Filler (made up of vegetable fibre, viscose fibre and diatomaceous earth)	1.0
Hexalene glycol	6.0
Colloidal silica	0.5

Tackifier (Acrylate copolymers available under the trade names Amphomer and AMP)		0.83
Surfactant (Teric LA8 from ICI)		0.3
Latex (60% aqueous solution)		1.06
Latex stabiliser	(Ammonia 0.06 Xanthan gum 0.06 $MgAlSiO_x$ 0.08)	0.20
Ethyl alcohol		20.0
Sodium benzoate		0.12
Lecithin		0.03
Silicone 346		0.05
Water		to 100%

The composition was shaken briskly for 30 seconds and then tested for viscosity in the Brookfields viscometer referred to earlier, at 20°C. The average viscosity of three tests was 5 cps.

A series of wheel spins balance tests were performed as follows.

A new tubeless Radial tyre (Kelly Steel Mark II P165/75 SR13) was supplied mounted on a rim and inflated. The wheel was checked for balance, deflated and 150ml of the composition of EXAMPLE II was inserted via the valve cock with the valve removed. The wheel was inflated to 241kps, checked for balance and as it was within 20g, weights were not corrected. The wheel was fitted to the right rear of a passenger vehicle.

A new tubeless Radial tyre (Kelly Steel Mark II P165/75 SR13) was supplied with a tube fitted, mounted on a rim and inflated. The wheel was balanced (30g inner 65g outer), deflated and 150ml of the composition of EXAMPLE II was inserted via the valve cock with the valve removed. The wheel was inflated to

248kps, checked for balance and as it was within 30g, weights were not corrected. The wheel was fitted to the right front of a passenger vehicle.

To demonstrate the puncture sealing effectiveness of the composition of EXAMPLE II, a test was conducted by employing the passenger vehicle fitted with the tyres referred to in the preceding paragraphs.

A spike plate containing five steel spikes (9mm in diameter, 30mm high which were ground to a point) was positioned to puncture the tread area of the wheels containing the composition of the invention. The vehicle was driven over the spike plate at low speed and then continued on for approximately 11km at varying speeds of up to 100km/h on a Proving Ground circuit.

An inspection of the punctured tyres at the end of the test drive revealed that there was no detectable loss in tyre pressure and no visible leak when the wheels were placed in a water bath. The tyre fitted with the tube was disassembled and the tube removed, the tube had five puncture holes in it.

EXAMPLE III

The components of the composition of EXAMPLE II were modified to replace the Xanthum Gum with an equal amount by weight of a mixture of two polyuronites and calcium oxalate.

The composition was sent to the Mechanical Laboratory of the Laboratories and Services Division of the Australian Army Engineering Development Establishment and tested as follows.

In this test a current model passenger vehicle, Commodore Executive, VR, sedan was fitted with Goodyear Eagle GA tubeless tyres of the size P205/65 R15. The procedure involved in the test was as follows.

The tyres were removed from the vehicle and balanced on a dynamic wheel balance machine. The tyre pressure was checked and inflated to 32psi. The vehicle was driven on the Proving

Ground First Class circuit for a distance of 14km, during this test phase, speeds of approximately 120km/h were reached with no detectable vibration at the steering wheel.

The tyres were fully deflated and 120ml of the composition of EXAMPLE III inserted via the valve stem. The tyres were then inflated to 32psi, the vehicle was driven on the Proving Grounds First Class circuit for a distance of 14km at speeds approximately 170km/h with no detectable vibration at the steering wheel. A distance of 21km was travelled on a public road to confirm that no vibration was present at the steering wheel.

To determine if the balance had been altered each wheel was removed from the vehicle and mounted on the dynamic wheel balance machine. The results were;

a. Left front wheel, ... outer 10grams,
..... inner ok,
b. Right front wheel, ... outer 8-10grams,
..... inner 8grams,
c. Right rear wheel, ... outer 8-10grams,
..... inner ok,
d. Left rear wheel ..... outer ok,
..... inner ok.

NOTE: No weights were required or added to adjust the balance of the wheels.

A spike plate consisting of five (5), 8mm diameter steel rods which were ground to a point, 30mm long and welded upright to a flat steel plate was used to puncture the tyres. The spike plate was positioned 1/3 of the tyre width in from the outer tyre wall of the left front wheel and the vehicle was driven over it and then 2/3 of the tyre width in from the outer tyre wall of the left rear wheel and the vehicle was driven over it.

The spike plate was re-positioned 1/3 of the tyre width in from the outer tyre wall of the right front wheel and the vehicle was

driven over it, then 2/3 of the tyre width in from the outer tyre wall of the right rear wheel and the vehicle was driven over it. With all tyres spiked the vehicle was driven on the Proving Ground First Class circuit for a distance of 17km reaching speeds of approximately 170km/h. A short distance of the rough course variable pitch corrugations and the fixed pitch corrugations were traversed to simulate the impact of rough road driving.

The tyre pressures were checked and recorded at 34psi(hot). The right front wheel was removed from the vehicle and placed in a water bath, no leak was detected. The tyre was demounted from the rim and five (5) puncture holes were identified from the inside of the tyre. With the solution still in the tyre, the tyre was remounted to the rim, re-inflated to 32psi before being submerged in the water bath. It was seen that two (2) of the puncture holes were leaking, but after rotating the wheel for approximately three revolutions the punctures had sealed. Each wheel was placed in the water bath and all punctures were sealed.

Testing of the composition of EXAMPLE III was also conducted in England and the Schedule adopted for testing purposes was ECE Regulation 30 (drum test).

ECE Regulation 30 is a load/speed performance simulation of a tyre under load running on the road. The test consists of an inflated tyre and wheel assembly, mounted on a free running axle, with the tyre pressed against a power driven rotating drum. Through the axle a force related to the particular tyre's operating load is applied to the drum which is then rotated at varying speeds for specified periods of time.

PROCEDURE:

(a) Two new tyres size: 205 70 VR 14 Michelin XWX Tubeless were fitted to appropriate rims, inflated to 30 psi and balanced.

(b) The valve cores were removed and 100ml of a composition of the invention was applied through each valve.

(c) Valve cores were replaced and the tyres re-inflated to 30 psi.

(d) Each tyre was rebalanced - one tyre gave zero deviation (4 checks); the second tyre gave an imbalance of only 15gms to the outer fitted side only (4 checks)

NOTE: *The balance of a competitive tyre sealant product was similarly tested. The variable imbalance readings of 35-65gm were recorded (4 checks).*

(e) Both tyres were fitted to the front hubs of a Mercedes 500 SEL. Each tyre was then driven over a length of pre-fabricated steel plate containing eight 30mm vertical spikes. The diameter of the spikes at the point of crown penetration through the tyre's inner tubeless liner was measured at 5mm.

This is represented diagramatically in Fig. 1 of the drawings.

(f) The car was then driven over normal roads for a distance of 30 miles. One of the tyres was removed from the vehicle for the purpose of testing to ECE Regulation 30.

(g) Prior to testing, a 5 mm diameter nail was hammer driven at three separate points through the tyre's tread and removed.

The penetrations, one centre, one to the left and one to the right were spaced at approximately one-third intervals around the tyre's circumference.

This is represented diagramatically in Fig. 2 of the drawings.

(h) Air loss was evident at each point of 'fabricated' penetration. As the tyre was rotated by hand, each penetration sealed within seconds.

(i) The tyre inflation pressure was adjusted in accordance with the ECE Regulation 30 drum test requirement for 'V' speed rating (150 mile/hour - 240 Km/h).

The fitted assembly was mounted on the test rig:

Test Details: **(Using 1.7mm diameter drum)**

Schedule: 10 minutes to 200 Kph
10 minutes at 200 Kph
10 minutes at 210 Kph
10 minutes at 220 Kph
20 minutes at 230 Kph

(equivalent to 150 mile/hour - 240 Km/h)

Load Applied - 502.32 Kg

Bay Temperature at Start of Test - 22°C

Bay Temperature at End of Test - 26°C

Inflation Pressure at Start of Test - 44 psi

Inflation Pressure at End of Test - 56 psi

THE TYRE COMPLETED THE ABOVE TEST SCHEDULE

(j) Following removal of the tyre assembly from the test rig, two 3mm diameter nails and two 5mm diameter nails were hammer driven through the crown area of the tyre and removed. **The tyre was hand rotated after each penetration and air loss was sealed in seconds.**

(k) The tyre/rim was refitted to the vehicle and driven over normal roads for a further 30 miles.

(l) The tyre/rim was removed from the vehicle in order to re-check the balance. The maximum imbalance recorded after four checks was 10gm.

(m) The valve core and one bead were removed in order to visually check the viscosity of the material. The viscosity of the product after drum testing was unaffected.

(n) With valve core and bead replaced; the tyre was inflated to 30 psi and the assembly refitted to the vehicle.

CONCLUSIONS: **(After completion of Tests)**

(i) Pressure loss: There was no measurable loss of tyre inflation pressure.

(ii) Viscosity: The viscosity of the product appeared unaffected.

(iii) Balance: The balance was satisfactory.

In spite of **FIFTEEN** penetrations sustained to the test tyre - 8 spiked; 3 nail penetrations prior to ECE testing and 4 nail penetrations after ECE testing - the Tyre Seal product performed and functioned effectively as a tyre sealant both on the road and throughout the duration of ECE Regulation 30 test schedule conducted to 150 mile/hour - 240 Km/h.

EXAMPLE IV

A composition was formulated for use in high speed tyres, as follows.

	Percentage by weight
Filler (made up of Viscose fibre 0.5mm in length, diatomaceous earth, vegetable fibre and comminuted rubber of 400 micron average diameter)	1.75
Hexylene glycol	2.50
Ethylene glycol	4.50
Colloidal silica	1.15
Tackifier (Acrylic resin RL 3034	2.00
Surfactants (Teric LA8 and Teric 12A3 from ICI)	0.30
Lecithin / Linseed Oil	0.30
Polyuronate and calcium oxalate	0.12
Rust inhibitor / Preservative (Sodium benzoate)	0.07
Water	to 100%

The composition was subjected to the same wheel spin balance test referred to earlier.

120ml of the composition of EXAMPLE IV was introduced into a P205/65/R15 tubeless vehicle tyre and the wheel spun on a balancing machine. The test was conducted several times and showed an out of balance reading from zero to 7gm over the tests. A reading of 7 qm does not require correction and is considered to be an acceptable reading for in balance. This compares with readings of 26 gram inside and 31 gram outside out of balance determined for an existing commercially available composition which produced noticeable out of balance when the tyre was being used.

The out of balance readings in a more severe test, the rapid spin balancing start up test, were 15 gram per wheel for the composition of this invention and 54 grams for the existing commercially available composition. As will be appreciated by the person skilled in the art, the 15 gram out of balance would generally be considered not to be a noticeable unbalancing effect. The 54 gram unbalance would be considered as substantially out of balance.

To demonstrate the puncture sealing effectiveness of the composition of EXAMPLE IV the tyre was subjected to similar treatment as referred to in relation to the composition of EXAMPLE III as conducted by the Australian Army.

At the end of the test no appreciable air loss was detected from the tyre.

In a further test another similar tyre containing 120ml of the composition of the invention was inflated to 2.1 kpa (32 psi) and punctured by driving four 30mm X 2mm nails through the tread. The tyre was spun after each puncture.

The tyre was then placed on a free spinning axle. A driven drum with an uneven surface to simulate a rough road, was provided and the tyre moved against and held against the drum under pressure. The drum was rotated, to rotate the tyre, at up to speeds equivalent to 130 km/hour vehicle speed.

The drum test was continued for 48 hours and the air pressure in the tyre checked at the end of that time. No appreciable air loss was detected from the tyre.

The tyre was then fitted to a motor vehicle and driven under normal road conditions. After 3 days the air pressure was checked and again, no appreciable air loss was detected from the tyre.

CLAIMS

1. A composition comprising 0.7 to 5% by weight filler; 0.5 to 15% silica in a colloidal form; 4 to 30% by weight of a glycol; 1 to 8% by weight tackifier; up to 5% by weight surfactant in the event that the tackifier does not exhibit surfactant properties; with the balance being water; said composition having a working viscosity (as hereinbefore defined) of less than 20 cps.

2. A composition as claimed in claim 1 wherein the filler is selected from one or more of synthetic fibres; vegetable fibres; mineral particles; and, comminuted rubber particles.

3. A composition as claimed in claim 1 or claim 2 wherein the glycol is an alkyl or alkylene glycol of 5 to 30 carbon atoms.

4. A composition as claimed in any one of the previous claims wherein the tackifier is selected from one or more of the group of stabilised latex solutions; PVA polymers; acrylic polymers; and acrylamide / acrylate copolymers.

5. A composition as claimed in claim 1 further including up to 20% lower alcohol and/or up to 3% lecithin or linseed oil.

6. A composition as claimed in claim 1 wherein the colloidal silica is a mixture of hydrophobic and hydrophilic fumed silica.

7. A composition as claimed in any one of the preceding claims wherein the working viscosity is between 2 and 10 cps.

8. A composition as claimed in claim 1 further including up to 5% by weight of a mixture of two or more polyuronites either with or without trace amounts of calcium oxalate.

9. A method for preventing loss of air from pneumatic tyres as a result of a puncture therein; comprising providing within said tyre, or within an inner tube in said tyre if present, a puncture preventative amount of a composition as claimed in any one of the preceding claims.

10. A method as claimed in claim 9 wherein the tyre is an automobile tyre and the puncture preventative amount is between 120 ml and 150 ml.

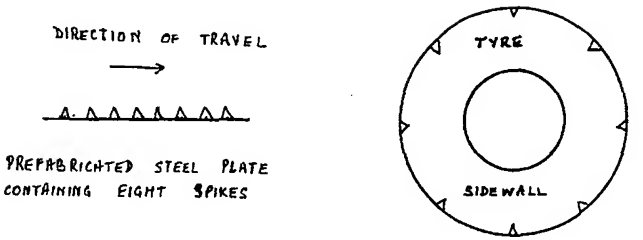
DIRECTION OF TRAVEL
PREFABRICATED STEEL PLATE CONTAINING EIGHT SPIKES
TYRE
SIDEWALL

FIG 1

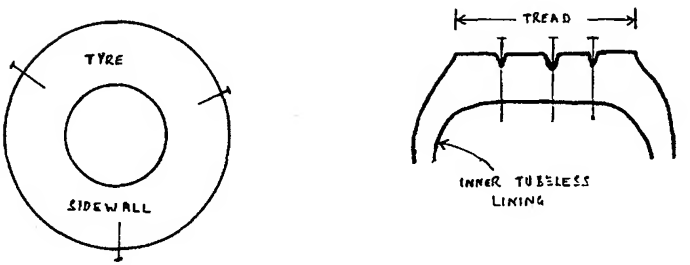
TYRE
SIDEWALL
TREAD
INNER TUBELESS LINING

FIG 2

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/AU 95/00487

A. CLASSIFICATION OF SUBJECT MATTER

Int Cl[6]: B29C 73/22, 73/16, 73/02, C09K 3/10, 3/12 // B29L 30:00, 23:24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC : B29C 73/02, 73/16, 73/22, C09K 3/10, 3/12, B60C 21/08, 21/14

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
AU : IPC as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
DERWENT : silica: and glycol and IPC as above
JAPIO : silica: and glycol and IPC as above

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3167522 A (SHULMAN) 26 January 1965 column 1 lines 40-43, Example	1-10
A	US 2760542 A (PETERSON et al.) 28 August 1956 whole document	1-10
A	US 2286963 A (HOUSER et al.) 16 June 1942 whole document	1-10

[X] Further documents are listed in the continuation of Box C

[X] See patent family annex

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"&" document member of the same patent family

Date of the actual completion of the international search
03 november 1995

Date of mailing of the international search report
14 NOVEMBER 1995

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INTERNATIONAL SEARCH REPORT

International Application No.
PCT/AU 95/00487

C (Continuation) **DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 796908 A (THE GENERAL TIRE & RUBBER COMPANY) 18 June 1958 whole document	1-10
A	EP 0074791 A (STAUFFER CHEMICAL COMPANY) 23 March 1983 page 1 lines 4-7, claim 8	1-10
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No.
PCT/AU 95/00487

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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